



Guide for Conducting Treatability Studies under CERCLA: Solvent Extraction

Office of Emergency and Remedial Response
Hazardous Site Control Division OS-220

QUICK REFERENCE FACT SHEET

Section 121 (b) of CERCLA mandates EPA to select remedies that "utilize permanent solutions and alternative treatment technologies or resource recovery technologies to the maximum extent practicable" and to prefer remedial actions in which treatment that "permanently and significantly reduces the volume, toxicity, or mobility of hazardous substances, pollutants, and contaminants is a principal element." Treatability studies provide data to support remedy selection and implementation. They should be performed as soon as it becomes evident that the available information is insufficient to ensure the quality of the decision. Conducting treatability studies early in the remedial investigation/feasibility study (RI/FS) process should reduce uncertainties associated with selecting the remedy and should provide a sound basis for the Record of Decision (ROD). Regional planning should factor in the time and resources required for these studies.

This fact sheet provides a summary of information to facilitate the planning and execution of solvent extraction remedy screening and remedy selection treatability studies in support of the RI/FS and the remedial design/remedial action (RD/RA) processes. Detailed information on designing and implementing remedy screening and remedy selection treatability studies for solvent extraction is provided in the "Guide for Conducting Treatability Studies Under CERCLA: Solvent Extraction," Interim Guidance, EPA/540/R-92/016a, August 1992.

INTRODUCTION

There are three levels or tiers of treatability studies: remedy screening, remedy selection, and remedy design. The "Guide for Conducting Treatability Studies Under CERCLA: Solvent Extraction," Interim Guidance, discusses all three levels of treatability studies. The solvent extraction treatability guidance is one of a series of technology-specific documents.

Remedy screening studies provide a quick and relatively inexpensive indication of whether solvent extraction is a potentially viable remedial technology. Remedy selection studies provide data that permit evaluation of solvent extraction's ability to meet expected site cleanup goals and provide information in support of the detailed analysis of the alternative (i.e., seven of the nine evaluation criteria specified in EPA's RI/FS Interim Final Guidance Document, EPA/540/G-89/004, 1988). Remedy selection tests generally have moderate costs and may require weeks to months to complete. Remedy design testing provides quantitative performance, cost, and design information for remediating the operable unit. Remedy design studies are of moderate to high costs and may require months to complete.

TECHNOLOGY DESCRIPTION AND PRELIMINARY SCREENING

Technology Description

Solvent extraction is a separation technology which uses a fluid to remove hazardous contaminants from excavated soils, sludges, and sediments and/or contaminated groundwater and surface water. The solvent is chosen such that the contaminants have a higher affinity for the solvent than for the contaminated material. Solvent extraction does not destroy contaminants, it concentrates them so that they can be recycled or destroyed more cost effectively. When contaminants are not recycled, solvent extraction must be combined with other technologies in a treatment train to destroy the separated, concentrated contaminants. Solvent extraction has limited application as a treatment technology for inorganic contaminants. Nevertheless, solvent extraction may affect inorganic contaminants even when the process is designed to treat organic contaminants. The discussions in this document are primarily related to organic contaminants.

Solvent extraction processes can be divided into three general types based upon the type of solvent used:

standard solvents, near-critical fluids/liquefied gases, and critical solution temperature (CST) solvents. Standard solvent processes use alkanes, alcohols, or similar liquid solvents typically at ambient pressure. Near-critical fluid/liquefied gas processes use butane, isobutane, propane, carbon dioxide (CO₂), or similar gases which have been liquefied under pressure at ambient temperature. Systems involving CST solvents use the unique solubility properties of those compounds to extract contaminants at one temperature where the solvent and water are miscible and then separate the concentrated contaminants from the water fraction at another temperature. Solvent is then removed from the contaminants by evaporation.

Figure 1 is a general schematic of the solvent extraction process.

Feed preparation (1) includes moving the material to the process where it is normally screened to remove debris and large objects. Depending upon the process vendor and whether the process is semi-batch or continuous, the waste may need to be made pumpable by the addition of solvent or water. In the extractor (2), the feed and solvent are mixed, resulting in the dissolution of organic contaminant into the solvent. The extracted organics are removed from the extractor with the solvent and go to the separator (3), where the pressure or temperature is changed, causing the organic contaminants to separate from the solvent. The solvent is recycled (4) to the extractor, and the concentrated contaminants (5) are removed from the separator.

Solvent extraction has been used as a full-scale remedy at two CERCLA sites: (1) the Treban PCB site in Tulsa, OK and (2) the General Refining site in Garden City, GA. However, the technology shows promise for treating a variety of organic contaminants commonly found at CERCLA sites. During fiscal year 1989, solvent extraction was selected in combination with other technologies for remediation of five Superfund sites having soils and sediments contaminated with poly-chlorinated biphenyls (PCBs), polynucleararomatic hydrocarbons (PAHs), pentachlorophenol (PCP), and other organic compounds. These sites are Norwood PCBs, MA; O'Conner, ME; Pinette's Salvage Yard, ME; Ewan Property, NJ; and United Creosoting, TX.

Prescreening Characteristics

The determination of the need for and the appropriate tier of treatability study required is dependent on the literature information available on the technology, expert technical judgement, and site-specific factors. The first two elements - the literature search and expert consultation - are critical factors of the prescreening phase in determining whether adequate data are available or whether a treatability study is needed.

Information on the technology applicability, the latest performance data, the status of the technology, and sources for further information are provided in one of a series of engineering bulletins being prepared by U.S. EPA's Risk Reduction Engineering Laboratory (RREL) in Cincinnati, Ohio.

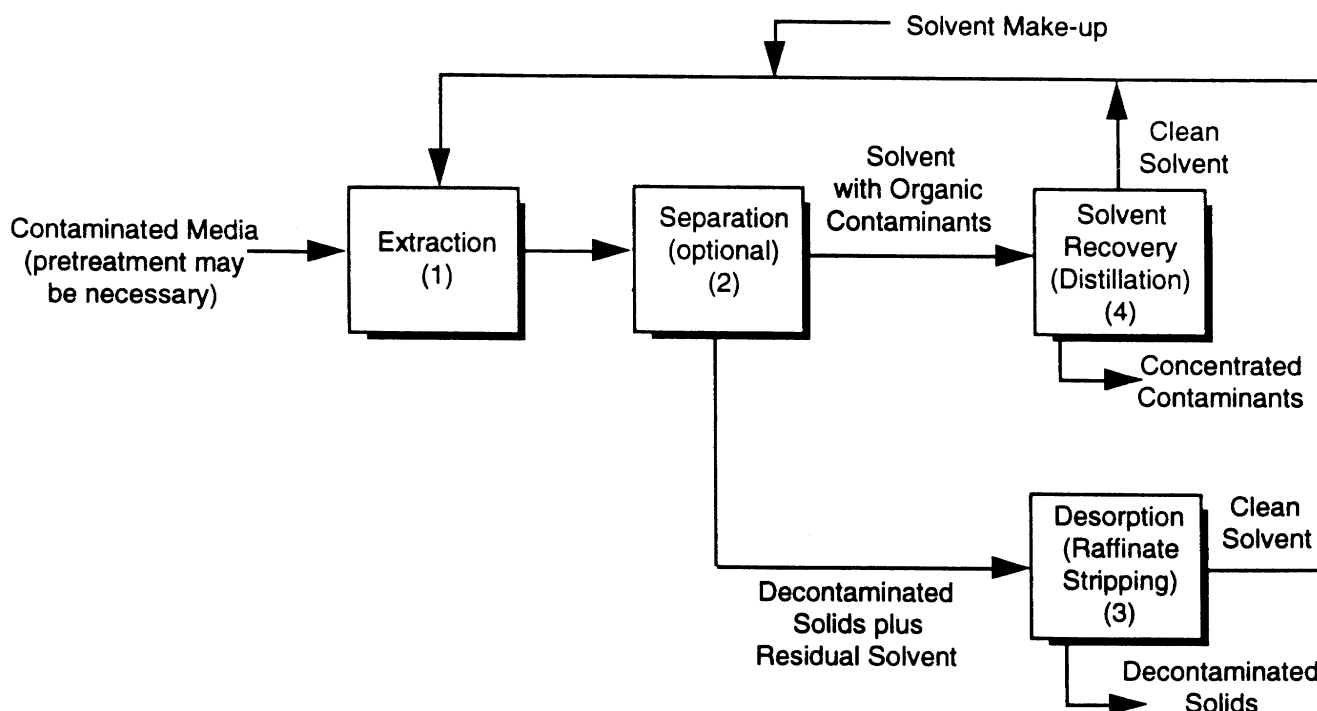


Figure 1. Generic Solvent Extraction Process

A literature search should be performed to determine the physical and chemical properties of the contaminants of interest. The most important prescreening parameters are the contaminant profile and concentration of contaminants. Contaminant characteristics such as vapor pressure, solubility, Henry's Law constant, partition coefficient, boiling point, specific gravity, and viscosity may be important for the design of remedy evaluation studies and related residuals treatment systems. Tests for total organic carbon (TOC) and total recoverable petroleum hydrocarbons give an estimate of equilibrium partitioning and contaminant transport between soil and water and may be useful in comparing results to other sites with different contaminants. Particle size distribution and moisture are useful for evaluating materials handling and pretreatment processes. A discussion of other, less important parameters such as pH, temperature, chemical oxygen demand (COD), and contaminant toxicity is contained in the solvent extraction guide.

If contamination exists in different soil strata or in different media, a characterization profile should be developed for each soil type or media. Available chemical and physical data (including contaminant concentration averages and ranges) and the volumes of the contaminated soil requiring treatment should be identified. For "hot spots", separate characterizations should be done so they can be properly addressed in the treatability tests. Solvent extraction may be applicable to some parts of a site, but not to other parts.

The quantity of large rocks, debris, and other oversize screenable material that must be removed is an important measurement. While this is not a "laboratory" measurement, it is important to determine which treatment method is most suitable for preparing the bulk soil or sediment for entry into the solvent extraction process, i.e., screening to remove large rocks, stumps, debris, and washing or crushing of oversize materials, etc. The quantity of and degree of contamination of water is important for design of ultimate treatment systems. The water could be the media to be treated or could be associated with a soil/sludge media.

Technology Limitations

Solvent extraction limitations may be defined as characteristics that hinder cost-effective treatment of the contaminated media with specific processes. The limitation may be due to the contaminant (incompatibility with the selected solvents or complex mix of contaminants), the process, or the media. Several extraction stages may be required in some cases to meet the site cleanup goals. Difficulties may be encountered in recycling spent solvents. Hydrophobic and hydrophilic contaminants may be difficult to extract with the same solvent. The contaminated media might require substantial pretreatment.

Complex mixtures of contaminants in the waste media, such as a mixture of metals, non-volatile organics, semi-volatile organics, etc. may make the design or selection of a suitable solvent extraction system that will remove all the different types of contaminants difficult. Organically bound

metals can co-extract with the target organic pollutants and restrict disposal and recycle options. The presence of emulsifiers and detergents can adversely affect the extraction performance by competing with the extraction solvent for retention of the organic pollutants. High moisture can interfere with the efficiency of some solvents, limiting the application of certain solvent extraction processes.

Advantages and disadvantages exist among the various types of solvent extraction processes. The primary differences include the following: ability to handle fines or high clay content, ability to handle a wide variety of organic contaminants, the ease of phase separation after extraction, and the energy requirements.

THE USE OF TREATABILITY STUDIES IN REMEDY EVALUATION

Treatability studies should be performed in a systematic fashion to ensure that the data generated can support the remedy evaluation process. The results of these studies must be combined with other data to fully evaluate the technology.

There are three levels or tiers of treatability studies: remedy screening, remedy selection, and remedy design. Some or all of the levels may be needed on a case-by-case basis. The need for and the level of treatability testing are management-based decisions in which the time and cost of testing are balanced against the risks inherent in the decision (e.g., selection of an inappropriate treatment alternative). These decisions are based on the quantity and quality of data available and on other decision factors (e.g., state and community acceptance of the remedy, new site data).

Technologies may be evaluated first at the remedy screening level and progress through the remedy selection to the remedy design level. A technology may enter, however, at whatever level is appropriate based on experience with the technology, contaminants of concern, and site-specific factors. Figure 2 shows the relationship of three levels of treatability study to each other and to the RI/FS process.

Remedy Screening

Remedy screening, the first tier of testing, is used to determine the ability of a technology to treat a contaminated soil using simple laboratory tests. Approximately 5 kg of sample are extracted for several hours in a rotary shaker or other device using a hydrophilic solvent such as acetone or methanol. The residual solids are then extracted in a hydrophobic solvent such as hexane or kerosene. The mean contaminant concentration in the solids or water fraction is determined from duplicate samples before and after extraction. These studies are generally low cost (e.g., < \$30,000) and usually require one or more days to complete. Remedy screening tests are generic and can be performed at any laboratory with the proper equipment and qualified personnel. This tier is occasionally skipped for evaluation of solvent extraction.

Remedy Selection

Remedy selection, the second tier of testing, is used to evaluate the technology's performance on a contaminant specific basis for an operable unit. A total of 5 kilograms or more of sample are extracted, typically using vendor specific solvents and equipment. The test design is based on remedy screening tests or information from the prescreening search. Normally, triplicate samples are taken from both the solvent and the extracted medium (soil, water, etc.) These studies generally have moderate costs (e.g., \$20,000 to \$120,000) and may require several months or more to plan, obtain samples, and execute. They yield data that verify the technology's ability to meet expected cleanup goals and provide information in support of the detailed analysis of alternatives in the CERCLA Feasibility Study (FS).

The remedy selection tier of solvent extraction testing consists of bench-scale tests and/or pilot tests. Typically, these tests will be vendor-specific. Sufficient experimental controls are needed such that a quantitative mass balance can be achieved. The key question to be answered during

remedy selection testing is whether the treated media will meet the cleanup goals for this site. The exact removal efficiency or acceptable residual contaminant level specified as the goal for the remedy selection test is site-specific. Typically, a remedy design study would follow a successful remedy selection study, after the ROD.

Remedy Design

Remedy design testing is the third tier of testing. In this tier, pilot tests provide quantitative performance data to confirm the feasibility of solvent extraction based on target cleanup goals. These tests also produce information to refine cleanup time estimates and cost predictions and to design a full-scale system. This testing also produces the remaining data required to optimize performance. Specific information includes the identification of pretreatment requirements and material handling concerns and determining the number of extraction sequences required. These studies are of moderate to high cost (e.g., \$100,000 to \$500,000) and require several months to complete the testing. As with the other tiers, planning, analysis, and report writing will add to the duration of the study. For

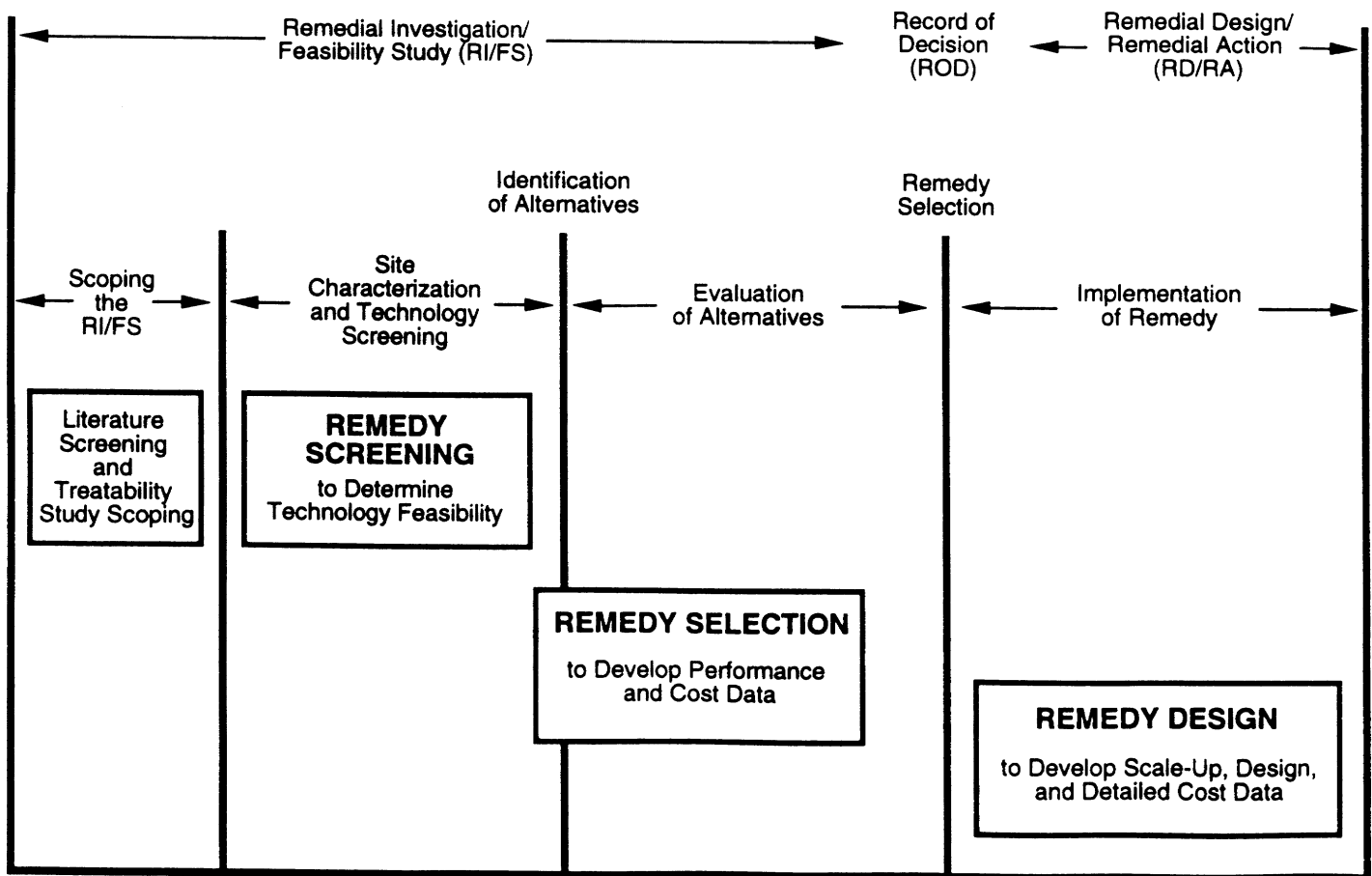


Figure 2. The Role of Treatability Studies in the RI/FS and RD/RA Process

complex sites (e.g., sites with different types or concentrations of contaminants in different media such as soil, sludges, and water), longer testing periods may be required, and costs will be higher. Remedy design tests yield data that verify performance to a higher degree than remedy selection and provide detailed design information. They are performed during the remedy implementation phase of the site cleanup, after the ROD and evaluation of alternatives.

TREATABILITY STUDY WORK PLAN

Carefully planned treatability studies are necessary to ensure that the data generated are useful for evaluating the validity or performance of the technology. The Work Plan sets forth the contractor's proposed technical approach to the tasks outlined in the RPM's Work Assignment. It also assigns responsibilities, establishes the project schedule, and estimates costs. The Work Plan must be approved by the RPM before work begins. A suggested organization of the solvent extraction treatability study Work Plan is provided in the "Guide for Conducting Treatability Studies Under CERCLA: Solvent Extraction".

Test Objectives and Goals

The overall solvent extraction treatability study objectives must meet the specific needs of the RI/FS. There are nine evaluation criteria specified in the EPA's RI/FS Interim Final Guidance Document. Treatability studies can provide data from which seven of these criteria may be evaluated.

Treatability study goals are the specific cleanup standards or removal rates designed to meet the test objectives. Setting goals for the treatability study is critical to the ultimate usefulness of its results. These goals must be well defined before the study is performed. Each tier or phase of the treatability study program requires appropriate performance goals. For example, remedy screening tests could answer the question, "Will solvent extraction reduce contaminants to the cleanup level, if known, or by a sufficient percentage (e.g., 50 to 70 percent)? The remedy selection tests measure whether the process could reduce contamination to below the anticipated performance criteria to be specified in the ROD. In the absence of specific cleanup goals, an arbitrary reduction (e.g., 90 to 99 percent) may be chosen to indicate potential usefulness.

Laboratory-scale tests are used for remedy screening. Remedy screening goals should simply require that the contaminant of interest shows a reduction in concentration in the soil of approximately 50 to 70 percent. The goal is to show solvent extraction has the potential to work at the site. Occasionally, sufficient information exists about soil conditions and contaminant solubility in various solvents so that remedy screening tests will not be necessary.

Bench-scale tests for remedy selection can determine if ultimate cleanup levels can be met. When solvent extraction is the primary treatment technology, the

suggested cleanup goals are typically set by the ARARs.

Pilot-scale testing occasionally is used during remedy selection. Pilot-scale tests usually involve the operation of a mobile treatment unit onsite for a period of 1 to 2 months. For more complex sites (e.g., sites with different types of contaminants in separate areas), longer overall testing periods may be required. The goal of pilot-scale testing is to confirm that the cleanup levels and treatment times estimated for site remediation are achievable.

Experimental Design

Careful planning of experimental design and procedures are required to produce adequate treatability study data. The experimental design must identify the critical parameters and determine the number of replicate tests necessary. System design, test procedures, and test equipment will vary among vendors. The information presented in this section provides an overview of the test equipment and procedures as these relate to each type of test.

Screening tests can be rapidly performed in onsite or offsite laboratories using standard laboratory glassware or specially designed laboratory-scale extractors to evaluate the potential performance of solvent extraction as an alternative technology. Typically, one or more hydrophobic and one or more hydrophilic solvents are tested. At this level of testing the experimental design should not be vendor-specific. Contaminant characteristics to examine during remedy screening include solubility in various solvents. Vapor pressure and Henry's Law constants are useful for evaluating solvent recovery methods. Observe whether an emulsion forms, either at the top or the bottom. Observe and time the solids settling rate and depth. The rate and the relative volume of the settling material will provide some indication of the potential for solids separation. Removal efficiency can be estimated by analyzing the separated solids for selected indicator contaminants of concern. It is usually not cost-effective to analyze for all contaminants at this level of testing. Check for other contaminants later in the solids or water fraction from remedy selection tests.

A remedy selection test design should be geared to the type of system expected to be used in the field (i.e., standard solvents, near-critical fluids/liquefied gases, or CST solvents). Solvent-to-solids ratios should be planned using the results from the laboratory screening tests, if they were performed. Remedy selection tests may use the same equipment as the remedy screening tests or may require that additional equipment be available, depending upon the process being evaluated. The tests are run under more controlled conditions than the remedy screening tests. The removal efficiency is measured under variable extraction conditions, which can include the addition of several solvents or an entrainer, heated solvents, pH adjustment, and use of supercritical or near-critical conditions. More precision is used in weighing, mixing, and phase separation. There is an associated increase in QA/

QC costs. Wet soils and sediments may require dewatering before treatment. Chemical analyses are frequently performed on the solvent fraction as well as on the cleaned solids fraction. Concentration measurements should be taken after each cycle or batch so that the cost of each cycle versus the percentage removal can be calculated and the impact of process variables on extraction efficiency can be quantified. This series of tests is considerably more costly than remedy screening tests, so only samples showing promise in the remedy screening phases should be carried forward into the remedy selection tier.

Bench-scale testing is usually sufficient for remedy selection, but there are instances where additional pilot-scale testing is warranted. If foaming problems occurred during remedy screening or bench-scale testing, pilot-scale testing should be used to solve any problems before full-scale remediation. Pilot-scale testing may be necessary in order to obtain community acceptance. A pilot-scale or short-term run with full-scale equipment may be used for large sites in order to better define cost estimates for the complete remediation.

The decision on whether to perform remedy selection testing on hot spots or composite samples is difficult and must be made on a site-by-site basis. Hot spot areas should be factored into the test plan if they represent a significant portion of the waste site. However, it is more practical to test the specific waste matrix that will be fed to the full-scale system over the bulk of its operating life. If the character of soils or sediments change radically (e.g., from clay to sand) over the depth of contamination, then tests should be designed to separately study system performance on each soil type.

SAMPLING AND ANALYSIS PLAN

The Sampling and Analysis Plan (SAP) consists of two parts—the Field Sampling Plan (FSP) and the Quality Assurance Project Plan (QAPjP). The RI/FS requires a SAP for all field activities. The SAP ensures that samples obtained for characterization and testing are representative and that the quality of the analytical data generated is known and appropriate. The SAP addresses field sampling, waste characterization, and sampling and analysis of the treated wastes and residuals from the testing apparatus or treatment unit. The SAP is usually prepared after Work Plan approval.

Field Sampling Plan

The FSP component of the SAP describes the sampling objectives; the type, location, and number of samples to be collected; the sample numbering system; the equipment and procedures for collecting the samples; the sample chain-of-custody procedures; and the required packaging, labeling, and shipping procedures.

Quality Assurance Project Plan

The QAPjP should be consistent with the overall objectives of the treatability study.

At the remedy screening level the QAPjP need not be overly detailed. The intended purpose of remedy screening tests is to determine if the contaminant concentration decreases by approximately 50 to 70 percent. Accurate calibration of the gas chromatograph with the target compounds is required. Duplicate tests are normally required at the remedy screening level to assure the reproducibility of the data.

The purpose of the remedy selection treatability study is to determine whether solvent extraction can meet cleanup goals and provide information to support the detailed analysis of alternatives (i.e., seven of the nine evaluation criteria). A higher level of QA/QC is required because the consequences of an incorrect decision are more serious at this level. Concentrations of the target contaminants in the soil should be verified by employing triplicate samples to provide a measure of data reproducibility. Recovery of contaminants from the sample is estimated by using matrix spikes. The QAPjP should address the measurement of critical variables, including the concentrations of target compounds in the initial and treated soil.

The methods for analyzing the treatability study samples are the same as those for chemical characterization of field samples. Preference is given to methods in "Test Methods for Evaluating Solid Waste," SW-846, 3rd. Ed., November 1986. Other standard methods may be used, as appropriate. Methods other than gas chromatography/mass spectroscopy (GC/MS) techniques are recommended to conserve costs when possible.

TREATABILITY DATA INTERPRETATION

To properly evaluate solvent extraction as a remediation alternative, the data collected during remedy screening and remedy selection phases must be compared to the test goals and other criteria that were established before the tests were conducted. In remedy screening treatability studies the contaminant concentration in the solids or water fraction before extraction is compared to the contaminant concentration in the same fraction after extraction. A removal of approximately 50 to 70 percent of the contaminants during the test indicates additional treatability studies are warranted. Before and after concentrations can normally be based on duplicate samples at each time period. The mean values are compared to assess the success of the study. Contaminant concentrations can also be determined for water and solvent fractions. However, these additional analyses add to the cost of the treatability test and may not be needed.

Remedy screening tests can sometimes be skipped when information about the contaminant solubilities in the selected solvent is sufficient to decide whether remedy selection studies will be useful. This information should be solvent- and contaminant-specific and may or may not be applicable to other sites.

In remedy selection, contaminant concentrations in the contaminated matrix before and after solvent extraction are typically measured in triplicate. Contaminant levels after treatment which meet site cleanup standards indicate solvent extraction has the potential to remediate the site. A reduction in the mean concentration to cleanup levels, if known, or by approximately 90 to 99 percent indicates solvent extraction is potentially useful in site remediation. A higher QA level is required with this tier of testing. A number of other factors must be evaluated before deciding to proceed with this technology to the evaluation of alternatives.

The design parameters for the solvent extraction process include material throughput and optimum solvent usage in gallons per dry ton of solids or gallons of water. It is important to estimate the volume and physical and chemical characteristics of each fraction to design treatment systems and estimate disposal costs. The ability to cost effectively recover used solvent is also important for cost and performance estimates. Removal efficiency measured as a function of the number of extraction stages can be used to determine the stages required to reach cleanup levels.

The final concentration of contaminants in the recovered (clean) solids fraction, in the solvents, in solvent distillation bottoms, and in water fractions are important to evaluating the feasibility of solvent extraction. The selection of technologies to treat the solvent or solvent still bottoms and water fraction from soil/sludges depends upon the types and concentrations of contaminants present. The amount of volume reduction achieved in terms of contaminated media is also important to the selection of solvent extraction as a potential remediation technology.

TECHNICAL ASSISTANCE

Additional literature and consultation with experts are critical factors in determining the need for and ensuring the usefulness of treatability studies. A reference list of sources on treatability studies is provided in the "Guide for Conducting Treatability Studies Under CERCLA: Solvent Extraction."

It is recommended that a Technical Advisory Committee (TAC) be used. This committee includes experts on the technology who provide technical support from the scoping phase of the treatability study through data evaluation. Members of the TAC may include representatives from EPA (Region and/or ORD), other Federal Agencies, States, and consulting firms.

OSWER/ORD operate the Technical Support Project (TSP) which provides assistance in the planning, performance, and/or review of treatability studies. For further information on treatability study support or the TSP, please contact:

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FOR FURTHER INFORMATION

In addition to the contacts identified above, the appropriate Regional Coordinator for each Region located in the Hazardous Site Control Division/Office of Emergency and Remedial Response or the CERCLA Enforcement Division/Office of Waste Programs Enforcement should be contacted for additional information or assistance.

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